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Standard Test Method for Vapor Pressure of Petroleum Products <u>and Liquid Fuels</u> (Mini Method—Atmospheric)¹

This standard is issued under the fixed designation D5482; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope Scope*

1.1 This test method covers a procedure for the determination of total vapor pressure of petroleum products <u>and liquid fuels</u> using automatic vapor pressure instruments. The test method is suitable for testing samples with boiling points above $\theta^{\circ}C$ (32°F)<u>0</u>°C (32°F) that exert a vapor pressure between 7 and 110 kPa (1.0 and 16 psi) at 37.8°C (100°F)<u>7 kPa and 110 kPa (1.0 psi and 16 psi) at 37.8°C (100°F)</u> at a vapor-to-liquid ratio of 4:1. The test method is applicable to gasolines containing oxygenates. No account is made of dissolved water in the sample.

Note 1—Because the external atmospheric pressure does not influence the resultant vapor pressure, this vapor pressure is an absolute pressure at $37.8^{\circ}C (100^{\circ}F) 37.8^{\circ}C (100^{\circ}F)$ in kPa (psi). This vapor pressure differs from the true vapor pressure of the sample due to some small vaporization of the sample and dissolved air into the air of the confined space.

1.1.1 Some gasoline-oxygenate blends may show a haze when cooled to $\theta \underline{0} \circ \underline{C}$ to $\underline{1} \circ \underline{C} \cdot \underline{1} \circ \underline{C}$. If a haze is observed in 8.5, it shall be indicated in the reporting of results. The precision and bias statements for hazy samples have not been determined (see Note 6).

1.2 This test method is a modification of Test Method D5191 (Mini Method) in which the test chamber is at atmospheric pressure prior to sample injection.

1.3 This test method covers the use of automated vapor pressure instruments that perform measurements on liquid sample sizes in the range from $\frac{11 \text{ mL}}{10 \text{ mL}}$ to $\frac{10 \text{ mL}}{10 \text{ mL}}$.

1.4 This test method is suitable for the determination of the dry vapor pressure equivalent (DVPE) of gasoline and gasoline-oxygenate blends by means of a correlation equation (see 13.2). The calculated DVPE is considered equivalent to the result obtained on the same material when tested by Test Method D4953.

1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.6 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety safety, health, and health environmental practices and determine the applicability of regulatory limitations prior to use. (For specific warning statements, see 7.2 through 7.7.)

<u>1.7 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.</u>

2. Referenced Documents

2.1 ASTM Standards:²

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

D4306 Practice for Aviation Fuel Sample Containers for Tests Affected by Trace Contamination

D4953 Test Method for Vapor Pressure of Gasoline and Gasoline-Oxygenate Blends (Dry Method)

*A Summary of Changes section appears at the end of this standard

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.08 on Volatility.

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For Annual Book of ASTM Standards volume information, refer to the standard's Document Summary page on the ASTM website.

D5190 Test Method for Vapor Pressure of Petroleum Products (Automatic Method) (Withdrawn 2012)³ D5191 Test Method for Vapor Pressure of Petroleum Products and Liquid Fuels (Mini Method) D5842 Practice for Sampling and Handling of Fuels for Volatility Measurement D5854 Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products

3. Terminology

3.1 *Definitions*:

3.1.1 dry vapor pressure equivalent (DVPE), n-value calculated by a defined correlation equation, that is expected to be comparable to the vapor pressure value obtained by Test Method D4953, Procedure A.

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3.1.2 gasoline-oxygenate blend, n-spark-ignition engine fuel consisting primarily of gasoline with one or more oxygenates.

3.1.3 *oxygenate*, *n*—oxygen-containing ashless organic compound, such as an alcohol or ether, which may be used as a fuel or fuel supplement. D4175

3.1.4 *platinum resistance thermometer, n*—temperature measuring device with platinum wire, whose electrical resistance changes in relation to temperature.

3.1.5 *total vapor pressure, n*—observed pressure measured in the experiment, that is the sum of the partial pressure of the sample and the partial pressure of the dissolved air.

3.1.6 *vapor pressure*, *n*—pressure exerted by the vapor of a liquid when in equilibrium with the liquid. D4175

3.2 Abbreviations:

3.2.1 DVPE, n-dry vapor pressure equivalent.

3.2.2 MTBE, n-methyl t-butyl ether.

4. Summary of Test Method

4.1 A known volume of chilled, air-saturated sample is introduced into a thermostatically controlled test chamber, the internal volume of which is five times that of the total test specimen introduced into the chamber. The test chamber is at atmospheric pressure prior to introduction of the sample. After introduction of the sample into the test chamber, the test specimen is allowed to reach thermal equilibrium at the test temperature, $37.8^{\circ}C$ (100°F). 37.8 °C (100°F). The resulting rise in pressure in the chamber is measured using a pressure transducer sensor and indicator.

4.2 The measured total vapor pressure is converted to a DVPE by use of a correlation equation (see 13.2).

5. Significance and Use

5.1 Vapor pressure is an important physical property of volatile liquids.

5.2 Vapor pressure is critically important for both automotive and aviation gasolines, affecting starting, warm-up, and tendency to vapor lock with high operating temperatures or high altitudes. Maximum vapor pressure limits for gasoline are legally mandated in some areas as a measure of air pollution control.

6. Apparatus

6.1 *Vapor Pressure Apparatus*—The type of apparatus⁴ suitable for use in this test method employs a small volume test chamber incorporating a transducer for pressure measurements and associated equipment for thermostatically controlling the chamber temperature.

6.1.1 The test chamber shall be designed to contain between $\frac{22 \text{ mL}}{20 \text{ mL}}$ and $\frac{50 \text{ mL}}{50 \text{ mL}}$ of liquid and vapor and be capable of maintaining a vapor-liquid ratio between 3.95 and 1.00 and 4.05 and 1.00.

6.1.2 The pressure transducer shall have a minimum operational range from 0 to 172 kPa (0 to 25.0 psi) <u>0</u> kPa to 172 kPa (0 psi to 25.0 psi) with a minimum resolution of 0.1 kPa (0.01 psi) <u>0.1</u> kPa (0.01 psi) and a minimum accuracy of ± 0.3 kPa (± 0.05 psi). ± 0.3 kPa (± 0.05 psi). The pressure measurement system shall include associated electronics and readout devices to display the resulting pressure reading.

6.1.3 A thermostatically controlled heater shall be used to maintain the test chamber at $\frac{37.8}{37.8}$ °C ± 0.1 °C (100 °F) ± 0.2 °F) 0.2 °F) for the duration of the test.

6.1.4 A platinum resistance thermometer shall be used for measuring the temperature of the test chamber. The minimum temperature range of the measuring device shall be from ambient to $75^{\circ}C$ (167°F) $75^{\circ}C$ (167°F) with a resolution of θ .1°C (θ .2°F) θ .1 °C (θ .2°F) θ .1 °C (θ .2°F) θ .1 °C (θ .2°F) θ .

³ The last approved version of this historical standard is referenced on www.astm.org.

⁴ The following instruments have been found satisfactory for use in this test procedure as determined by interlaboratory testing: Herzog Mini Reid Vapor Pressure Model MP970—available from Varlen Instruments, Inc., 2777 Washington Blvd., Bellwood, IL 60104 and ABB Model 4100—available from ABB Process Analytics, Lewisburg, WV. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.



6.1.5 The vapor pressure apparatus shall have provisions for introduction of the test specimen into the test chamber and for the cleaning or purging of the chamber following the test.

6.2 Syringe, if required, gas tight, $\pm 1 \text{ mL}$ to $20 \text{ mL} \pm 20 \text{ mL}$ capacity with a $\pm 1 \%$, or better, accuracy and a $\pm 1 \%$, or better, precision. The capacity of the syringe shall not exceed two times the volume of the test specimen being dispensed, and shall be chosen so as to provide maximum accuracy and resolution for the volume to be injected.

6.3 *Iced-Water Bath or Air Bath*, <u>Bath</u>, for chilling the samples and syringe to temperatures between $\Theta \circ C$ and $1 \circ C (321 \circ C)$ (32 $\circ F$ and $34 \circ F$). 34 $\circ F$).

6.4 *Pressure Measuring Device*, capable of measuring ambient and above ambient pressures with an accuracy of $\frac{0.20 \text{ kPa} (0.03 \text{ psi})}{0.20 \text{ kPa} (0.03 \text{ psi})}$ or better at the same elevation relative to sea level as the apparatus in the laboratory.

6.4.1 When a mercury manometer is not used as the pressure measuring device, the calibration of the pressure measuring device employed shall be periodically checked (with traceability to a nationally recognized standard) to ensure that the device remains within the required accuracy specified in 6.4.

6.5 *Pressure Source*, clean, dry compressed gas or other suitable compressed air capable of providing pressure for calibration of the transducer and cleaning of the cell.

NOTE 2-A vacuum source is an alternate means for cleaning of the cell.

7. Reagents and Materials

7.1 *Purity of Reagents*—Use chemicals of at least 99 % purity for quality control checks (see Section 11). Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.⁵ Lower purities can be used, provided it is first ascertained that the reagent is of sufficient purity to permit its use without lessening the accuracy of the determination.

7.2 Cyclohexane, (Warning—Cyclohexane is flammable and a health hazard).

7.3 Cyclopentane, (Warning—Cyclopentane is flammable and a health hazard).

7.4 2,2-Dimethylbutane, (Warning-2,2-dimethylbutane is flammable and a health hazard).

7.5 2,3-Dimethylbutane, (Warning—2,3-dimethylbutane is flammable and a health hazard).

7.6 2-Methylpentane, (Warning—2-methylpentane is flammable and a health hazard).

7.7 Toluene, (Warning—Toluene is flammable and a health hazard).

8. Sampling

8.1 General Requirements:

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8.1.1 The extreme sensitivity of vapor pressure measurements to losses through evaporation and the resulting changes in composition is such as to require the utmost precaution and the most meticulous care in the handling of samples.

8.1.2 Obtain a sample and test specimen in accordance with 10.3 of Practice D4057, D4177, D4306, D5842, or D5854 when appropriate, except do not use 10.3.1.8 of Practice the D4057, Sampling by Water Displacement, Displacement section for fuels containing oxygenates. Use a 1-L (1-qt) <u>1 L (1 qt)</u> sized container filled between 70<u>70</u> % to 80 % with sample.

<u>8.1.3</u> The present precision statement has been derived using samples in 1 L (1 qt) containers. Samples taken in containers of other sizes as prescribed in 8.1.2 can be used if it is recognized that the precision can be affected. In the case of referee testing, the 1 L (1 qt) sample container shall be mandatory.

8.1.4 Perform the vapor pressure determination on the first test specimen withdrawn from a sample container. Do not use the remaining sample in the container for a second vapor pressure determination. If a second determination is necessary, obtain a new sample.

8.1.5 Protect samples from excessive temperatures prior to testing. This can be accomplished by storage in an appropriate ice bath or refrigerator.

8.1.6 Do not test samples stored in leaky containers. Discard and obtain a new sample if leaks are detected.

8.1.7 Do not store samples in plastic (polyethylene, polypropylene, and so forth) containers since volatile materials may diffuse through the walls of the container.

8.2 Sampling Temperature—Cool the sample container and contents in an ice bath or refrigerator to the $\theta_0 \circ C$ to $1 \circ C (321 \circ C)$ (32°F to $34 \circ F$) $34 \circ F$) range prior to opening the sample container. Allow sufficient time to reach this temperature. Verify the sample temperature by direct measurement of the temperature of a similar liquid in a similar container placed in the cooling bath or refrigerator at the same time as the sample.

⁵ Reagent Chemicals, American Chemical Society Specifications, ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see AnnualAnalar Standards for Laboratory Chemicals, BDH Ltd., Poole, Dorset, U.K., and the United States Pharmacopeia and National Formulary, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.



8.3 Verification of Sample Container Filling—With the sample at a temperature of $\theta_0 \circ C$ to $1 \circ C, 1 \circ C, 1 \circ C$, take the container from the cooling bath or refrigerator and wipe dry with an absorbent material. If the container is not transparent, unseal it and using a suitable gage, confirm that the sample volume equals 7070% to 80% of the container capacity (see Note 3). If the sample is contained in a transparent glass container, verify that the container is 7070% to 80% full by suitable means (see Note 3).

8.3.1 Do not perform a vapor pressure test on the sample if the container is filled to less than $\frac{70}{70\%}$ by volume $\frac{\%}{70\%}$ of the container capacity.

8.3.2 If the container is more than $\frac{80\ \%}{10}$ by volume $\frac{\%}{10}$ -full, pour out enough sample to bring the container contents within the $\frac{7070\ \%}{10}$ to $\frac{80\ \%}{10}$ by volume $\frac{\%}{10}$ -range. Do not return any sample to the container once it has been withdrawn.

NOTE 3—For non-transparent containers, one way to confirm that the sample volume equals 7070% to 80% of the container capacity is to use a dipstick that has been pre-marked to indicate the 70 and 80% container capacities. The dipstick should be of such material that it shows wetting after being immersed and withdrawn from the sample. To confirm the sample volume, insert the dipstick into the sample container so that it touches the bottom of the container at a perpendicular angle, before removing the dipstick. For transparent containers, using a marked ruler or comparing the sample container to a like container that has the 7070% and 80% levels clearly marked, has been found suitable.

8.3.3 Reseal the container, if necessary, and return the sample container to the cooling bath or refrigerator.

8.4 Air Saturation of the Sample in the Sample Container:

8.4.1 Non-transparent <u>Containers</u>—<u>Containers</u>—With the sample again at a temperature between $\theta \underline{0} \circ \underline{C}$ and $\underline{1} \circ \underline{C}, \underline{1} \circ \underline{C}$, take the container from the cooling bath or refrigerator, wipe it dry with an absorbent material, remove the cap momentarily, taking care that no water enters, reseal, and shake it vigorously. Return it to the cooling bath or refrigerator for a minimum of 2 min.

8.4.2 *Transparent Containers*—Since 8.3 does not require that the sample container be opened to verify the sample capacity, it is necessary to unseal the cap momentarily before resealing it so that samples in transparent containers are treated the same as samples in non-transparent containers. After performing this task, proceed with 8.4.1.

8.4.3 Repeat 8.4.1 twice more. Return the sample to the bath or refrigerator until the beginning of the procedure.

8.5 Verification of Single Phase Samples—After drawing a test specimen and injecting it into the instrument for analysis, check the remaining sample for phase separation. If the sample is contained in a transparent container, this observation can be made prior to sample transfer. When the sample is contained in a non-transparent container, mix the sample thoroughly and immediately pour a portion of the remaining sample into a clear glass container and observe for evidence of phase separation. A hazy appearance is to be carefully distinguished from separation into two distinct phases. The hazy appearance shall not be considered grounds for rejection of the fuel. If a second phase is observed, discard the test and the sample. Hazy samples may be analyzed (see Report section).

9. Preparation of Apparatus

9.1 Prepare the instrument for operation in accordance with the manufacturer's instructions.

9.2 Prepare the sample introduction accessories, if required, in accordance with the manufacturer's instructions.

9.3 Chill the sample syringe to between $\theta 0 \circ C$ and $4.5 \circ C (32 \circ F) and <math>40 \circ F) 40 \circ F$ in a refrigerator or ice bath. Avoid water contamination of the syringe reservoir by suitably sealing the outlet of the syringe during the cooling process.

9.4 Clean and dry the test chamber in accordance with the manufacturer's instructions. With the test chamber sealed, observe that the instrument display is stable and does not exceed 0.00 ± 0.20 kPa $(0.00 \pm 0.03 \text{ psi})$. $0.00 \text{ kPa} \pm 0.20 \text{ kPa}$ $(0.00 \text{ psi} \pm 0.03 \text{ psi})$.

9.5 If in doubt of the cleanliness of the cell, refer to the cleaning procedure in the manufacturer's instructions, or if the display does not return to zero, refer to the calibration procedure in the manufacturer's instructions.

9.6 Verify that the temperature of the test chamber is within the required range from $\frac{37.8 \pm 0.1^{\circ}C}{(100 \pm 0.2^{\circ}F).37.8 \circ C \pm 0.1 \circ C}$ (100 °F ± 0.2 °F).

10. Calibration

10.1 Pressure Transducer:

10.1.1 Check the calibration of the transducer in accordance with the manufacturer's instructions on a monthly basis or when needed as indicated from the quality control checks (see Section 11). The calibration of the transducer shall be checked using two reference points, ambient barometric pressure, and a pressure above ambient pressure, determined by the operator, which is at least 80 % of the expected maximum pressure encountered during the test.

10.2 *Temperature Measuring Device*—Check the calibration of the temperature measuring device used to monitor the temperature of the test chamber at least every six months against a thermometer traceable to a national standard.

11. Quality Control Checks

11.1 Use a verification fluid with known volatility as an independent check against the instrument calibration each day the instrument is in use. For pure compounds (see 7.1), multiple test specimens may be taken from the same container over time,